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MATERIAL ASSESSMENT OF L97A1/L96A1 GRENADES FOR FOURIER TRANSFORM INFRARED SPECTROSCOPY AND THERMOGRAVIMETRIC ANALYSIS

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14. ABSTRACT

An effort was warranted to verify the material used the L97A1/L96A1 grenades due to a material deviation. The L97A1/L96A1 grenade drawings call for a 43% glass-filled nylon 6-12; however, it was discovered that a 30% glass-filled Nylon 6 may have been used instead. Fourier transform infrared spectroscopy and thermogravimetric analysis will be used to analyze the materials.

15. SUBJECT TERMS

Nylon 6 Nylon 6-12 Fourier transform infrared (FTIR) spectroscopy
Differential scanning calorimetry (DSC) L97A1/L96A1 grenades Thermogravimetric analysis (TGA)

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CONTENTS

FIGURES

SUMMARY

The Program Manager Close Combat Systems group tasked the Organics Materials Technology Branch, U.S. Army Armament Research, Development and Engineering Center, Picatinny Arsenal, New Jersey to investigate the current material used in the L97A1/L96A1 grenades. The current material is suspected to deviate from the material specification. The L97A1/L96A1 drawings call for a 43% glass-filled Nylon 6-12; however, through laboratory analysis, it was determined to be 30% glass-filled Nylon 6 may have been used instead.

Fourier transform infrared spectroscopy (FTIR) was also conducted on baseline samples of Nylon 6-12 and Nylon 6. The FTIR was also conducted on the samples taken from both old and the new L97A1/L96A1 grenades.

Thermogravimetric analysis (TGA) was conducted on both the old and the new L97A1/L96A1 grenades.

It was concluded that the old grenades contained the 43% glass-filled Nylon 6-12 as specified in the drawing and the new grenades contained 30% glass-filled Nylon 6.

INTRODUCTION

The L96A1 is an anti-riot grenade discharger, while the L97A1 is a practice anti-riot grenade discharger. During a teleconference on12 April 2010, it was discovered that the material used did not match the material specification for the L97A/L96A1 grenades. The call out on the drawing stated that 43% glass-filled Nylon 6-12 will be used. However, the material used was discovered to be a 30% glass-filled Nylon 6.

While the material in the datasheet was very similar, there were slight variations in the modulus of elasticity and the impact strength. Additionally, the Nylon 6 is more prone to water absorption issues compared to the Nylon 6-12, due to the increases in the amide/carbon ratio. Nylon 6 has water absorption of 9.5% at saturation while Nylon 6-12 has a water absorption rate of 1.6% at saturation. In addition, swelling due to water absorption causes the material to lose tensile strength. The amount of strength lost is relative to the amount of swelling.

In addition to water absorption, glass content adds to the mechanical property of the material. Glass-filled (43%) Nylon 6-12 will have greater mechanical properties compared to 30% glass-filled Nylon 6. Table 1 shows the difference in the properties of the two nylons from their data sheets.

Table 1
Properties of nylons with different glass content

Properties	30% Nylon 6	40% Nylon 6-12
Impact strength, Izod		
Notched 1/8 in. (3.2 mm) section	2.0 ft-lb/in.	2.7 ft-lb/in.
Unnotched 1/8 in. (3.2 mm) section	19.0 ft-lb/in.	22.0 ft-lb/in.
Tensile strength	23000 psi	25000 psi
Tensile elongation	3.0 - 4.0 %	3.0 - 4.0 %
Tensile modulus	1.35 x 10^6 psi	1.60 x 10^6 psi
Flexural strength	34000 psi	38500 psi
Flexural modulus	1.20 x 10^6 psi	1.40 x 10^6 psi

PROCEDURE

Materials

Old L96A1/L97A1 grenade (grey)
New L96A1/L97A1 grenade (grey)
Baseline Nylon 6 sample
Baseline Nylon 6-12 sample

Samples

Small fragments were taken from the old and new L96A1/L97A1 grenade (figs. 1 and 2). The specimens were then analyzed by methods of FTIR and TGA.

The FTIR is an analytical process that displays the major peaks of various functional groups of a sample. This test will be used to examine the differences in the chemical component. The FTIR will be performed on baseline Nylon samples and compared to the new and old grenade to identify the resins used in each grenade.

The TGA is an analytical test which determines the changes in weight in relation to change in temperature. For this test, samples from the old and new grenade are heated until the ash, the glass fiber, is the only material remaining.

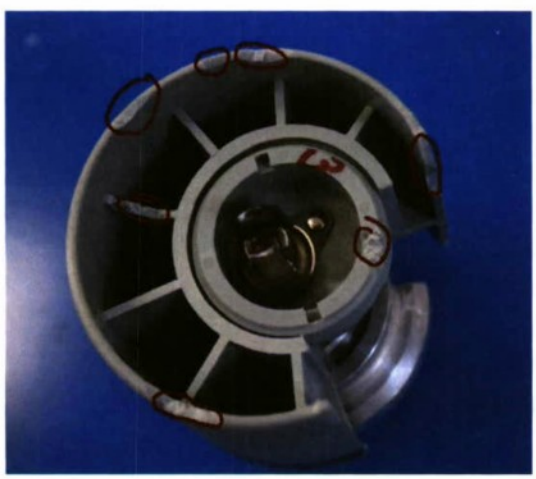

Figure 1
Old L96A1

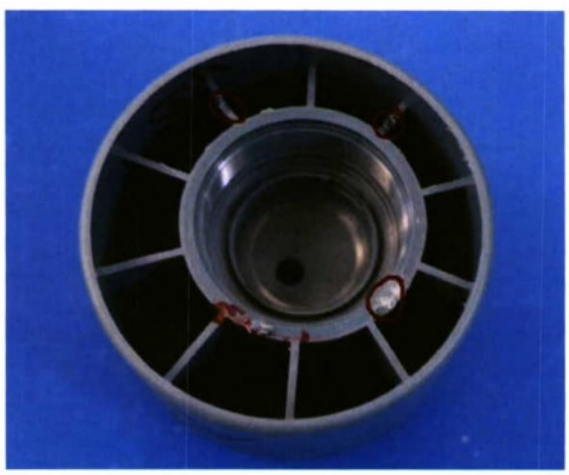

Figure 2
New L97A1

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy

The FTIR scan (fig. 3) shows two baseline materials

- The blue spectra represent the Nylon 6 sample
- The black spectra represent Nylon 6-12 sample

The relative size and location of the peaks are used to identify the material. The peak location determines which functional group is present while the size determines the amount present in the molecule. Amide is the main functional group in Nylon. The peaks in the spectra characterize this group can be found in table 2. The peak at 900 cm^{-1} is due to the bending of an Amide in the Nylon 6-12. The main difference between the two Nylons lies in its structure. Nylon 6 has an Amide group on every sixth carbon, while in Nylon 6-12 the Amide group alternates from being on the sixth carbon apart and twelfth carbon apart. The size of the major peaks in Nylon 6 is larger than in Nylon 6-12 due to the greater amide/carbon ratio.

Table 2
Major peaks of nylon

Major peaks (cm^{-1})	Bond type
3300	N-H
Twin peak at 2900 to 2800	C-H
1640	C-O
1545	C-N

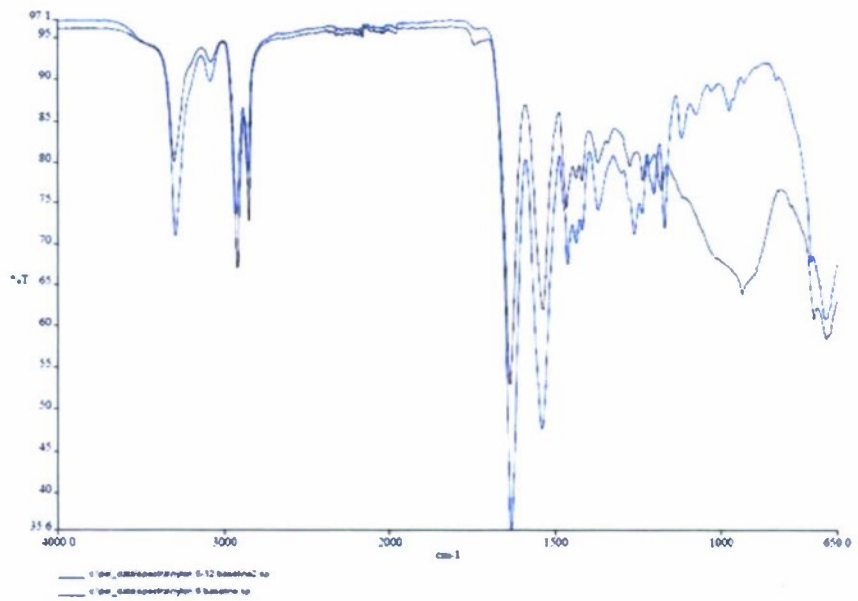

Figure 3
FTIR analysis of Nylon 6 versus Nylon 6-12

The FTIR scan (fig. 4) shows two materials.

- The blue spectra represent the baseline Nylon 6-12 sample
- The black spectra represent a sample taken from the body of the older grenade body
- The relative size and location of all the peaks are identical, proving a strong confidence that the material is a Nylon 6-12 resin

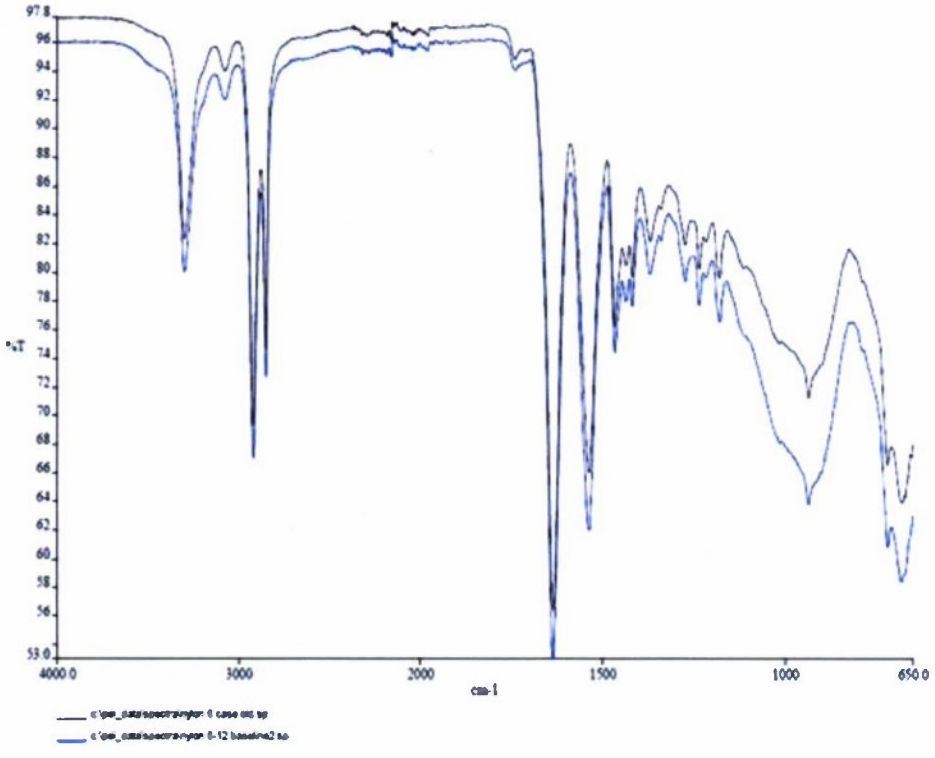

Figure 4
FTIR analysis of baseline Nylon 6-12 versus sample from old grenade body

The FTIR scan (fig. 5) shows two materials.

- The blue spectra represent the baseline Nylon 6 sample
- The black spectra represent a sample taken from the body of the blue (new) grenade body
- The relative size and location of all the peaks are identical, providing a strong confidence that the material is a Nylon 6 resin

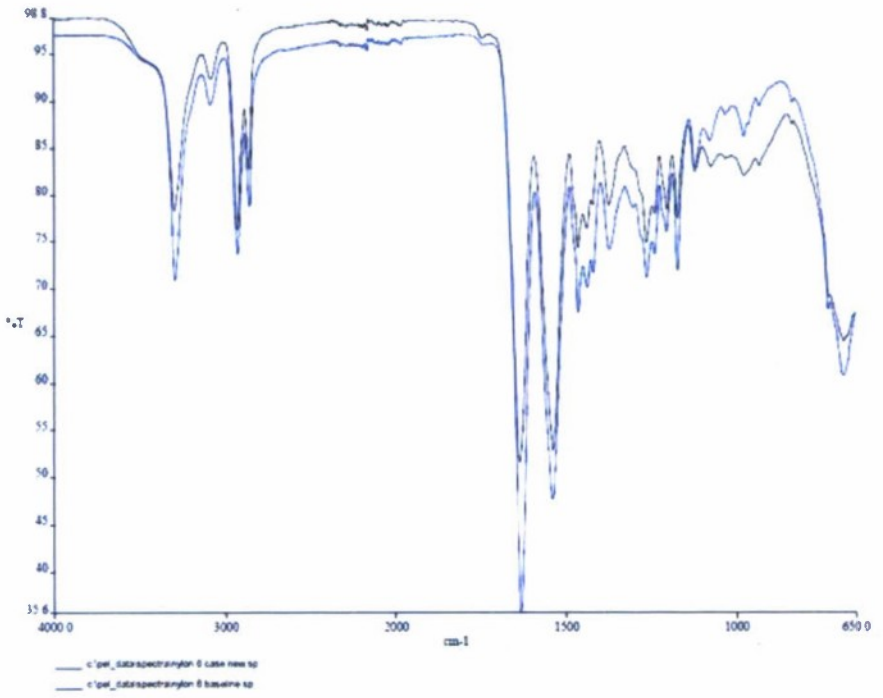

Figure 5
FTIR analysis of baseline Nylon 6 versus sample from new grenade body

Based on the findings of the testing, it can be concluded that the old grey grenade body was made from Nylon 6-12 (fig. 6) and the new blue grenade body was made from Nylon 6 (fig. 7).

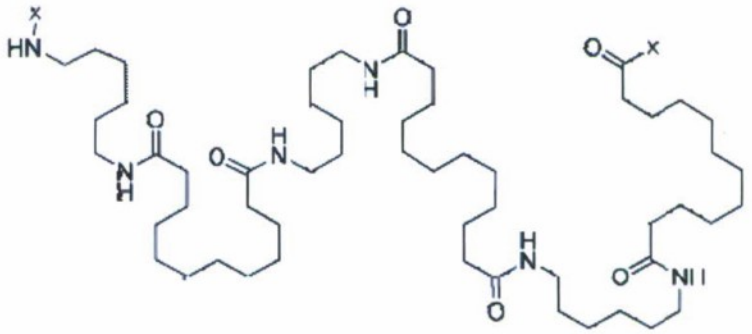

Figure 6
Nylon 6-12 structure

Figure 7
Nylon 6 structure

Thermogravimetric Analysis

The TGA is an analytical test that determines the changes in weight in relation to change in temperature.

The two materials were analyzed by TGA between the temperatures of 50 and 750°C.

There is virtually no weight loss between the temperatures of 50 and 200°C. So the weight loss is determined by using the temperature range of 100 to 700°C.

Temperatures between 600 to 700°C do not yield any additional weight loss. This information allows the analysis to be conducted between 100 and 700°C with confidence that no other weight loss will occur. The results are shown in table 3.

Table 3
TGA of sample from new and old grenade body

Residue remaining in percent	Run 1	Run 2	Run 3
New grenade	29.3	29.7	29.1
Old grenade	42.8	42.9	42.7

These results clearly indicate a discrepancy in the amount of residue remaining between heating the blue and grey material to high temperatures. The amount of material remaining represents the glass fill content of the materials, as glass melts at 2,300°C. As shown on the graph, the new grenade contained about 30% glass content, while the old grenade contained about 43% glass content.

CONCLUSIONS

Based on the results, the Fourier transform infrared spectroscopy and thermogravimetric analysis evidence indicated that there was a material switch in the L96A1/L97A1 grenades. Moreover, the results show that the new L96A1/L97A1 grenades were made with a 30% Nylon 6, while the old L96A1/L97A1 grenades were made with 43% Nylon 6-12.

Nylon 6 has a water absorption that is approximately 6.3 times greater than Nylon 6-12. When water is absorbed by the Nylons, it tends to swell, which may cause the material to become out of tolerance in some of the key critical areas. Swelling will also cause the material to lose tensile strength.

The Nylon 6 in the new grenades contained 30% glass fibers while the old grenades were made of Nylon 6-12 and 43% glass fibers. The lower glass content in Nylon 6 leads the material to have lower mechanical properties compared to the Nylon 6-12. Moreover, the swelling caused by water absorption will lower the tensile strength of Nylon 6. The lower mechanical properties may cause the material to fail more easily.

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